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TECHNICAL REPORT

PR-6

A GUARDED HOT-PLATE APPARATUS FOR THE MEASUREMENT OF THERMAL CONDUCTIVITY

QUARTERMASTER RESEARCH & ENGINEERING CENTER

PIONEERING RESEARCH DIVISION

OCTOBER 1962

NATICK, MASSACHUSETTS

AD- Div. 7 Accession No.

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Harold J. Hoge, Ph. D.

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Reference:
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October 1962

FOREWORD

This is the first of three reports now in preparation, which describe work in the field of thermal conductivity performed jointly by the Pioneering Research Division and the former Environmental Protection Research Division of this Center. The present report deals with the design, construction, and operation of the apparatus, and with the results of measurements made on a standard sample. Most of this work was performed by the Thermodynamics Laboratory of PRD.

The second report deals with construction and operation of the automatic control system with which the temperature of the guard ring of the thermal conductivity apparatus is held at the temperature of the hot plate. Most of this work was performed by the Biophysics Branch of EPRD.

The third report deals with experimental measurements made on a sample of standard underwear material (50 wool-50 cotton). Effects of variation in sample temperature, sample density, water content, nature of the surrounding gas, and pressure of the surrounding gas were studied. This work was performed jointly by the Biophysics Branch and the Thermodynamics Laboratory.

The work described in the second and third reports was supported in part by the Thermalibrium Suit program.

S. DAVID BAILEY, Ph. D.
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Pioneering Research Division

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Brigadier General, USA
Commanding
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CONTENTS

ABSTRACT

The design, construction, and operation of a guarded hot-plate apparatus for measuring thermal conductivity are described. The outstanding feature of the apparatus is its versatility. It has provision for the evacuation of the sample chamber and the control of pressure within it. The atmosphere surrounding the sample may be air or any other desired gas. Sample thickness may be adjusted or measured at any time without opening the sample chamber. Provision is made for future installation of strain gages with which to measure the force applied to the sample. Results of measurements made on a standard sample are presented, and compared with results obtained by the National Bureau of Standards on the same material. It is believed that the apparatus is capable of measurements accurate within 1 percent.

A GUARDED HOT-PLATE APPARATUS FOR THE MEASUREMENT OF THERMAL CONDUCTIVITY

1. The apparatus

a. General description

The apparatus is of the guarded hot-plate type. It uses a single sample, and has a top shield to prevent heat loss on the side of the hot plate where there is no sample. The circular hot plate is surrounded by an annular guard ring that prevents lateral heat flow to or from the hot plate.

The apparatus was designed for maximum versatility. It has provision for controlling the atmosphere surrounding the sample, by evacuation and if desired by the introduction of some gas other than air. Water vapor can also be introduced. The sample thickness can be measured or adjusted at any time without disturbing the progress of the measurements. The design is such that strain gages for measuring the compressive force on the sample could be installed if needed. The hot plate and guard ring are kept at the same temperature by automatic control.

Figure 1 is a photograph of the entire apparatus ready for use. It shows the sample chamber installed in the water bath, the control equipment including the potentiometer and power supply, and the refrigerating unit. Figure 2 is a close-up photograph of the sample chamber after removal from the water bath. Figure 3 is a scale drawing of the sample chamber, showing the hot-plate assembly and other essential parts of the apparatus.

The standards adopted by the American Society for Testing Materials (1) for the construction and use of the guarded hot plate were consulted while the apparatus was being designed, but it was apparent that some deviations from these standards would have to be made if the apparatus were to be as versatile as desired.

The unique feature of the apparatus is the provision for measuring and controlling most of the parameters affecting thermal conductivity while the sample is in place and even while measurements are in progress. None of the features of the apparatus are new in themselves, but the authors are not aware of any other apparatus that provides the same degree of versatility.

As mentioned in the Foreword, this report is the first of a group of three related papers. Additional information about the apparatus will be found in the second and third reports of the series (2, 3).

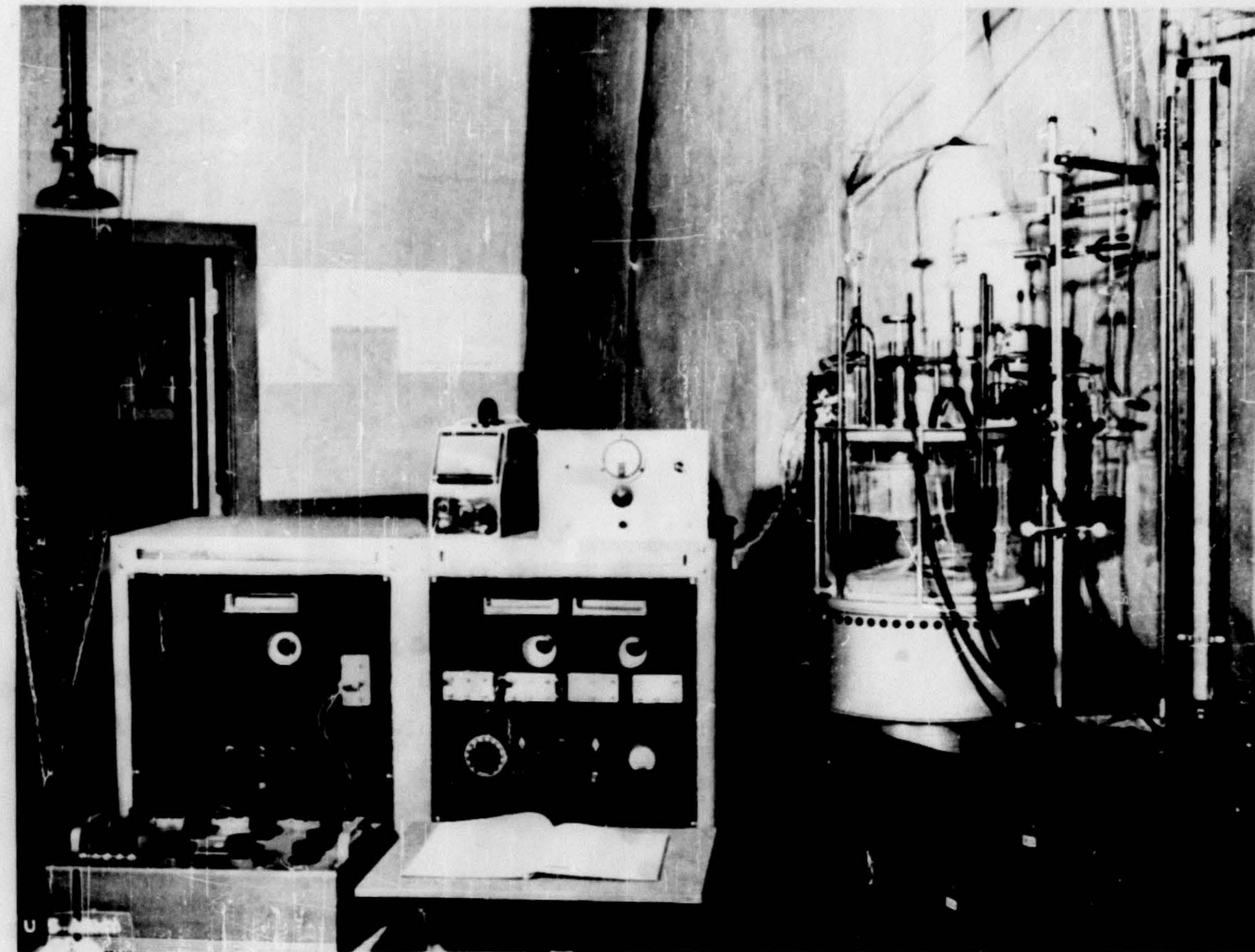


Fig. 1. Photograph showing the apparatus, and the measuring and control equipment used with it.

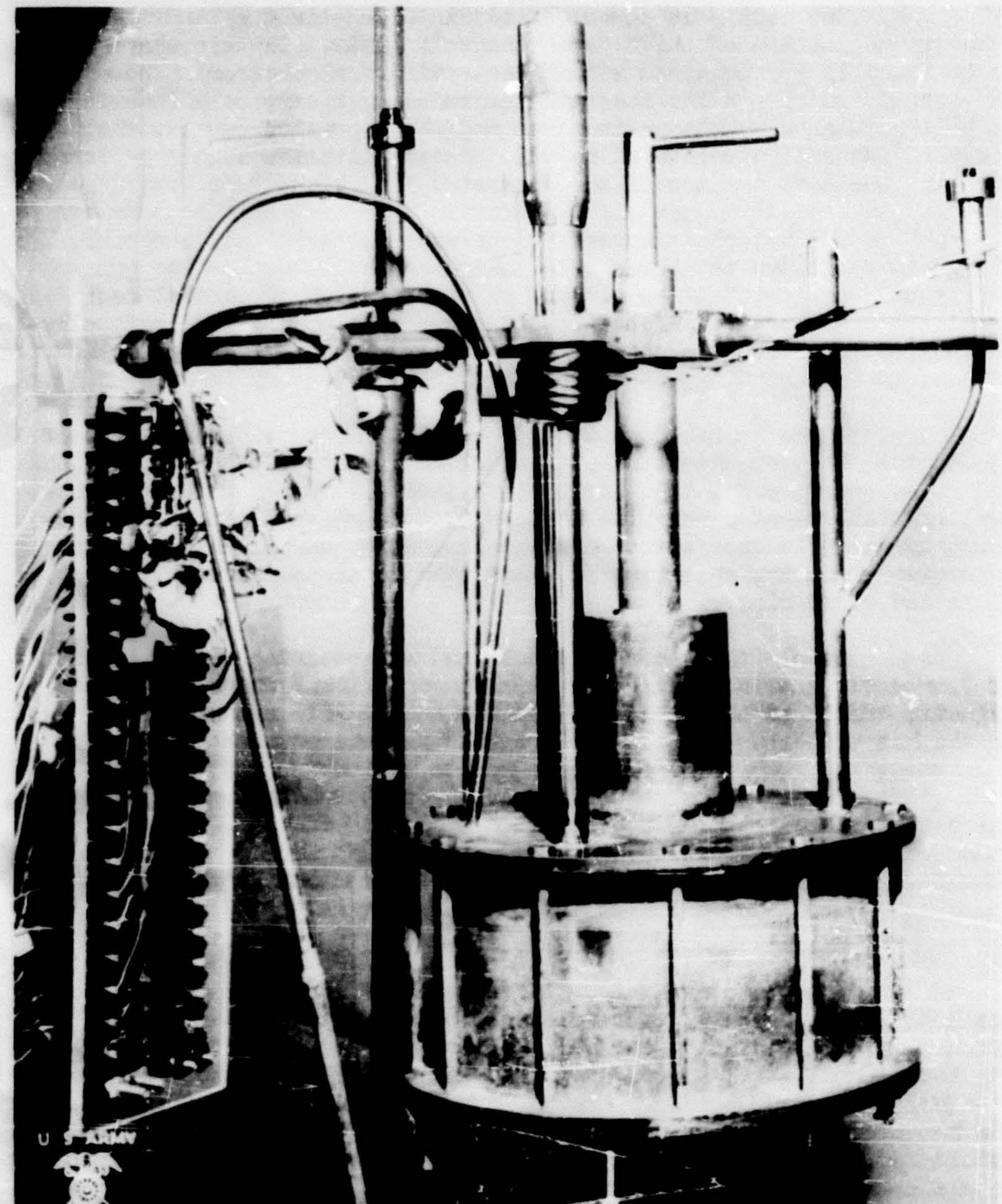


Fig. 2. Close-up photo of the guarded hot-plate apparatus. The thickness-measuring micrometer rests on one of the three tubes at which thickness measurements were made.

The sample is contained within a gas-tight cylindrical chamber, the bottom of which forms the cold plate. The circular hot-plate assembly is contained within the cylindrical chamber. The chamber may be opened for inserting or removing a sample by removing 12 bolts. When a sample is in place and the apparatus closed, the hot-plate assembly is lowered to make contact with the sample by turning the hand wheel at the top of the apparatus.

The hot-plate assembly consists of the hot plate proper, the guard ring, and the top shield. Each of the three has its own separate electrical heating circuit. The power for the hot plate is furnished by a Sorensen "Nobatron" DC power supply. The automatically-controlled power for the guard ring comes from the same source. The top shield is heated by manually-controlled AC.

Three copper-constantan thermopiles are used. Equality of temperature between the hot plate and guard ring is maintained with the aid of a 6-power pile, the output of which is fed to an automatic controller. Another 6-power pile is used for the measurement of ΔT, the temperature difference across the sample. A 3-power pile is used in maintaining the top shield at the same temperature as that of the hot plate and guard ring.

Sample thickness is determined by measuring the positions of 3 iron slugs with a differential transformer that responds to the magnetic effect of the slugs. The slugs are rigidly fixed with respect to the hot plate.

The sample chamber is connected to a vacuum system, with which the gas surrounding the sample can be pumped out. Fresh air or other gases can be admitted to the sample chamber through another connection.

b. Details of construction

Hot-plate assembly. The top shield forms the support for the hot plate and the guard ring. Nylon spacers, cut from 1/2-inch nylon rod, hold the hot plate and guard ring at a fixed distance below the top shield. Each spacer has a 1/4-inch diameter threaded projection on either end. One projection is screwed into a threaded hole in the hot plate or guard ring; the other passes through a hole in the top shield and is held by a nut. There are 3 spacers to hold the hot plate in place and 3 to hold the guard ring.

The hot plate and guard ring were made from a single piece of 3/8-inch sheet copper. This was first turned to the outside diameter of the guard ring (6.569 inches). Then with a pointed lathe tool a V-shaped circular slot was cut in one face of the plate, so as to separate the guard ring from the hot plate. The cutting was stopped just before the two parts were separated, and the groove was filled with General Electric Glyptal lacquer No. 1201-B. At a later stage in the construction, after the heaters and thermocouples had been installed, the plate was fastened to the top shield with the Glyptal groove toward the shield. The assembly was then mounted in a lathe and on the exposed side about 0.070 inch of the copper plate was faced off. In this operation the copper remaining at the base of the Glyptal groove was cut away and the hot plate and guard ring became separate parts mounted coaxially, with a ring of Glyptal of trapezoidal cross section filling the space between. At the surface that makes contact with the sample, the space filled with Glyptal has a width of 0.018 inch. At the other surface the width is much greater.

The scheme of construction described above worked very well the first time, but it was necessary to remove the hot plate and guard ring from the top shield several times, to rewind heaters, relieve thermocouple shorts, and the like. After several reassemblies, the hot plate and guard ring surfaces were found to be not quite coplanar and some straightening was required. It might help to use heavier nylon spacing plugs.

The 5/16-inch space between the top shield and the hot plate or guard ring was loosely filled with cotton batting. This reduced the injurious effect of small undesired departures of the shield from the desired temperature. It also reduced the error that might result from any nonuniformity of top-shield temperature. The space above the top shield, between it and the top of the sample chamber, was also filled with cotton batting during some of the runs. The operation of the apparatus is also improved by this cotton, but it is not needed unless the highest accuracy is involved, and its use undoubtedly delays the approach to the steady state when the redistribution of water vapor is involved.

The diameter of the hot plate, measured to the middle of the Glyptal spacer, is 3.482 inches, whence the width of the guard ring is 1.544 inches.

The top shield is a circular brass plate 1/4 inch thick, with an integral downward-projecting lip at the periphery that prevents any direct radiation from outside the shield from falling on the hot plate.

Adjustment of hot-plate position. Rotation of the hand wheel at the top of the apparatus causes the supporting shaft to move up or down and raises or lowers the hot-plate assembly. A large Sylphon bellows permits the motion to be transmitted into the gas-tight sample chamber from outside. At the lower end of the supporting shaft is a ball-and-socket joint through which force is transmitted from the rod to the hot-plate assembly. The joint housing carries 3 radial spring-steel cantilever arms, the outer ends of which are attached to the top shield with nylon-plug spacers.

The object of the ball-and-socket joint is to allow the hot plate and guard ring to conform to a sample that may not be distributed over the bottom of the sample chamber with complete uniformity (for example, when granules, powder, or other loose material is being measured). It also allows the supporting shaft to rotate with respect to the hot-plate assembly. The joint has caused some trouble, however, because it permits the hot-plate assembly to tip when the edge of the guard ring catches on the wall of the sample chamber. This happens often enough so that it is doubtful whether the ball-and-socket joint is worth the trouble it causes.

Provision for force measurement. The three cantilever arms referred to above are designed to have strain gages mounted on their surfaces, with which the force applied to the hot-plate assembly can be measured. This feature of the apparatus has not yet been used.

Sample chamber. The cold plate forms the bottom of the sample chamber. It is made from the same 3/8-inch copper sheet that is used for the hot plate and guard ring. The wall of the chamber is formed by a brass tube of 6.754-inch inside diameter, lined with a layer of 1/16-inch cork to give thermal protection to the edge of the guard ring. The top of the chamber is formed by a brass plate 3/8 inch thick. This plate and the cold plate were made thick enough so that the calculated flexure, with atmospheric pressure outside the sample chamber and zero pressure inside, does not distort the sample appreciably.

The joint between the top of the chamber and the chamber wall is sealed by a neoprene gasket, clamped by 12 bolts that pass through the projecting flanges of the top of the chamber and the cold plate. The surfaces between which the gasket is clamped have projecting ridges as recommended by van Heerden (4). Leaks at the gasket have given no trouble, although leaks elsewhere in the apparatus have caused considerable delay. When the bolts are removed, the sample chamber can be lowered from its lid for the insertion or removal of a sample.

Sample thickness. Three 1/2-inch monel tubes project above the top of the sample chamber. One of the three tubes is shown in Fig. 3. The tubes are soldered into holes in the chamber and are closed at

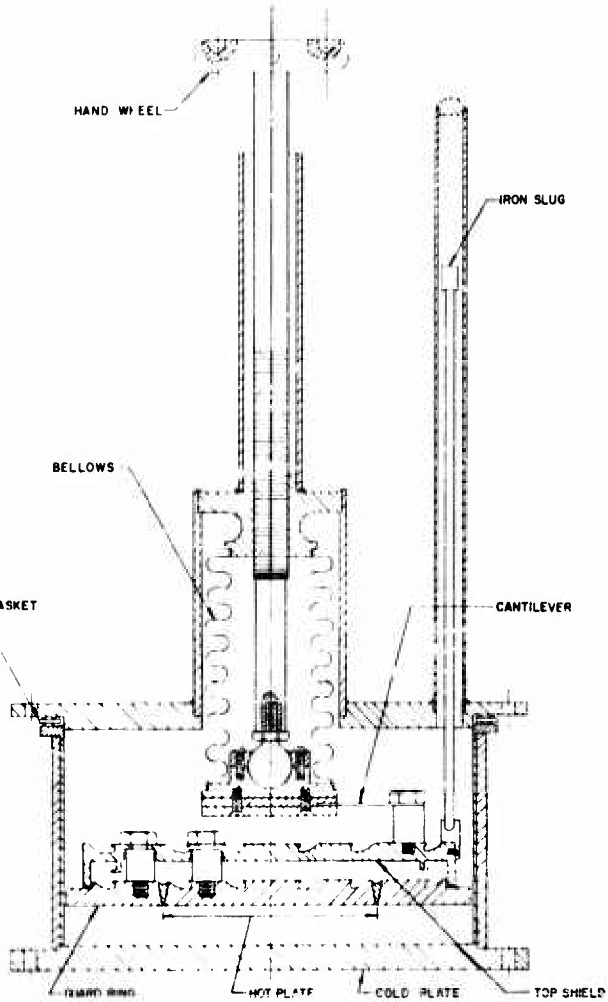


Fig. 3. Scale drawing of the sample chamber. Some details, such as electrical and vacuum connections, have been omitted to avoid confusion.

the top, forming extensions of the chamber. Each tube contains a smaller (1/8-in.) monel tube that carries an iron slug at its upper end and is rigidly fastened to the top shield at its lower end. The distance of the slug from the top of the tube in which it is housed is measured with a differential transformer mounted on a micrometer mechanism. The measuring device can be seen in Fig. 2, where it is sitting on one of the tubes that house the slugs, in position for making a measurement.

The two coils of the differential transformer form two arms of an AC bridge. They are wound side by side on a hollow spool that can be slipped over any one of the tubes in which the iron slugs are housed. To determine the position of a slug, the spool is slipped over the tube containing it and the micrometer is adjusted until the output of the bridge circuit is at a minimum. At this point the slug has a definite position approximately midway between the two windings of the differential transformer.

The three rods carrying slugs are equally spaced on the circumference of the top shield, and any nonuniformity of sample thickness is shown by differences in the three measurements. Calibration is required in order to relate readings of slug height to sample thickness. To calibrate, three steel blocks of known thickness are placed in the sample space, one just beneath each slug support. The apparatus is assembled, the over-all dimension of the main sample chamber is measured near each slug tube with a micrometer, and the position of each slug is measured with the transformer-micrometer device. The calibration is valid as long as the space occupied by the gasket is not changed. But if the clamping bolts are tightened, or if the apparatus is opened and reassembled, the over-all dimension of the sample chamber must be remeasured and the calibration data revised accordingly.

When a thickness measurement is made, three determinations of the position of each slug are made and averaged. The thickness at the point beneath each of the three slugs is computed and the values are compared. Assuming them to agree within about 0.010 inch, they are averaged and the average accepted as the sample thickness Δx.

Measurements of sample thickness are accurate to about 0.005 inch. The uncertainty in measuring slug position, if several readings are made, is about half this amount and could be improved without great difficulty.

Hot-plate power. The Sorensen power supply (Model E-28-5) furnishes up to 5 amperes at any voltage between about 23 and 31. It is used at a fixed setting near the rated value of 28 volts. The possibility of using such a power supply rather than storage batteries was suggested by the Heat Transfer Section of the National Bureau of Standards. The power supply is entirely satisfactory and has been a great convenience.

The circuits for controlling and measuring the hot-plate power are shown in Fig. 4. The current is adjusted to the desired value with the three variable series resistors, which give coarse and fine adjustment. The fixed resistor limits the maximum current to a safe value. The total current I + i is determined from the potential drop across the 1-ohm standard resistor with a Leeds & Northrup Type K-3 potentiometer.

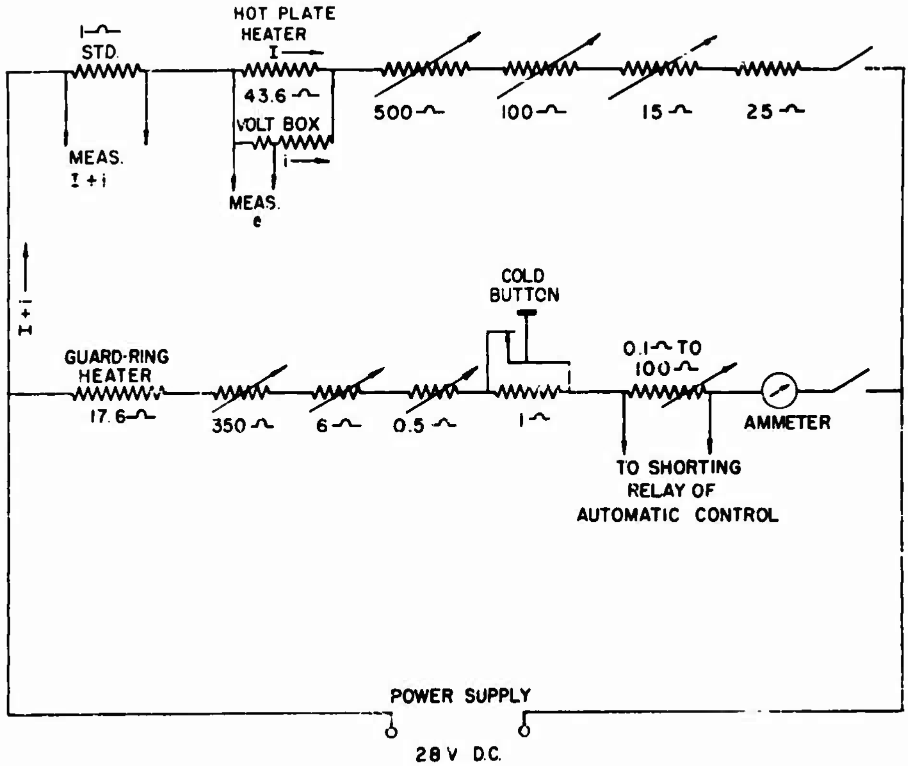


Fig. 4. Hot-plate and guard-ring power circuits, showing means of control and the arrangement for measuring hot-plate power.

The hot-plate heater winding is of the 4-terminal type, with the current and potential wire branch-points at the plate itself, so that the measured heat input does not include the heat developed in the connecting wires. The potential wires are connected to a volt box with a ratio of 100 to 1, and a total resistance of 15,000 ohms. The volt box is thus in parallel with the hot-plate heater, and permits the potential drop E across it to be determined from the potential drop e, which is small enough to come within the range of the Type K-3 potentiometer. The volt box ratio $r = E/e$ is slightly greater than 100 because of the two connecting wires between the box and the heater.

The current i in the volt box is calculated and subtracted from the total current to give the current I in the heater. Then the power supplied to the hot plate is calculated as EI. The resistance of the hot-plate heater does not appear explicitly in the computations. It varies somewhat with temperature.

Guard-ring power. Power for the guard ring is drawn from the same DC unit that supplies the hot plate. The circuit is shown in Fig. 4. The "cold button," which is useful in manual control, is a normally-closed shorting switch across a 1-ohm resistor in the power-control circuit. When depressed, this switch introduces the 1-ohm resistor into the circuit and reduces the guard-ring power slightly. Convenient control is obtained if the power is set at a level very slightly higher than that required for temperature constancy. Then, whenever the galvanometer shows the guard ring to be slightly too hot, the cold button is depressed to bring its temperature down.

Automatic control. Automatic control of the guard-ring power, to maintain it at the same temperature as the hot plate, is performed by a special controller described in the second of the three reports of this series. A relay actuated by the controller alternately opens and closes a short across a resistance, and thus produces small increases and decreases in power. The period of the relay is about 5 seconds.

Manual adjustment is made until the intervals of increased power and the intervals of lower power are each about 2 1/2 seconds long. When more power is required, the automatic controller increases the length of the intervals of higher power, and correspondingly decreases the intervals of slightly lower power. The size of the resistor that is periodically shorted by the relay can be varied from 100 ohms down to 0.1 ohm.

A large resistance, causing a large change in power when the relay is actuated, is appropriate when the steady state has not yet been reached. A small resistance is desirable after the steady state is reached, so that the opening and closing of the relay will not cause large temperature oscillations of the guard ring. A value of 0.5 ohm is satisfactory in the steady state. With this value the tendency to lose control is not troublesome, and yet the temperature oscillations can hardly be observed.

The relative lengths of the intervals of higher and lower power are varied continuously by the controller to correspond to the size of the temperature-error signal, and, since the period of the intervals is quite small compared to the response time of the guard ring, proportional control is obtained.

Top-shield power. Figure 5 shows the top-shield power circuit. The 115-volt AC supply is reduced to about 50 volts by the first Variac autotransformer. Fine adjustment is obtained with the second Variac and still finer adjustment with a 3-ohm variable resistor. A "cold button" is included, which when depressed completely opens the circuit.

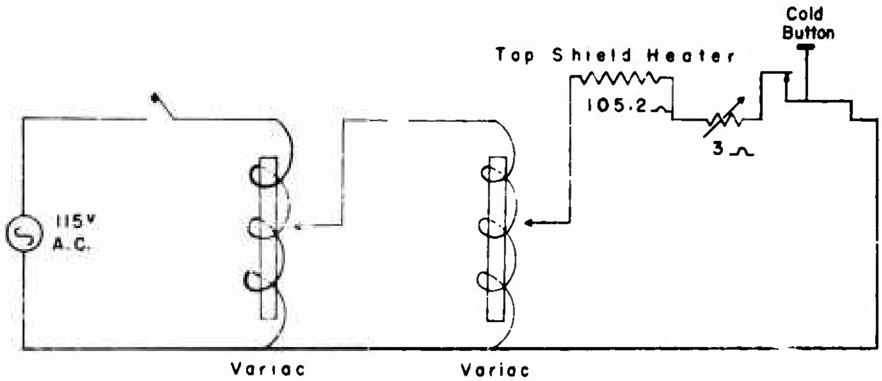


Fig. 5. Top-shield power circuit, showing means of control.

Operation might be improved if the top-shield power were taken from the same DC supply used for the hot plate and guard ring, but rewinding of the top-shield heater would be required, which up to the present has not been considered justified.

Temperature difference. There are three thermocouple systems used in the system of temperature control and measurement.

These systems are shown schematically in Fig. 6. This figure also shows the locations of all the junctions that are placed on the hot plate or guard ring. One of the three thermocouple systems is used to determine the temperature difference (Δ T) existing across the sample. This system is a 6-power copper-constantan thermopile with one set of junctions immersed in the water bath and the other set imbedded in the guard ring. In principle it would be better to imbed the junctions in the hot plate itself but this would require more wires to terminate on the hot plate, with a greater chance of undesired heat conduction. Therefore the guard ring is used as a reference and other temperatures are measured by comparison with its temperature.

The output of the thermopile is measured with the Type K-3 potentiometer, and divided by 6 to get the output per couple. The temperature of the water bath is measured with a mercury-in-glass thermometer. Entering the temperature-emf table of the thermocouple at this temperature, and going upward by the amount of the observed emf, the temperature of the guard ring is found, and the hot-plate temperature is taken to be equal to it.

The cold-plate temperature is taken to be equal to the water-bath temperature. A correction could be made for the temperature drop through the cold plate, but for the measurements made thus far the correction is small enough to be neglected. A small temperature drop between the cold plate and the bath is to be expected, but the bath was vigorously stirred in order to keep this drop negligible.

Thermopile between hot plate and guard ring. A second 6-power copper-constantan thermopile permits equality of temperature to be maintained between the hot plate and the guard ring. The pile is connected to a Leeds & Northrup Model 2430-A galvanometer and to a Brown Electronik Model 104W1G null detector. The two detecting instruments are in parallel. In the null detector the thermocouple voltage is chopped, amplified, detected, and passed through a zero-center indicating meter. The meter current is used to actuate the magnetic amplifier of the automatic control system.

The galvanometer is used principally for establishing the zero point at which the thermopile emf is zero. The galvanometer has a sensitivity of 2 mm per microvolt. When the control is functioning properly, the movement of the galvanometer spot is hardly detectable.

Thermopile between top shield and guard ring. A 3-power copper-constantan thermopile compares the temperature of the top shield with that of the guard ring. The emf of this pile is observed on a second Model 2430-A galvanometer, and is held at zero by manual control

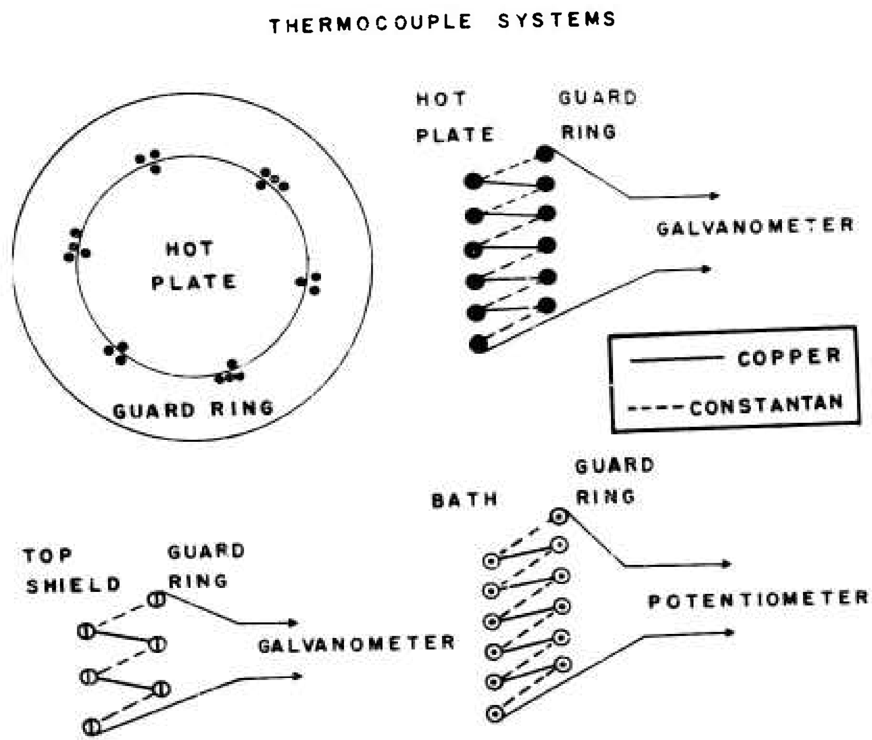


Fig. 6. Schematic representation of the three thermocouple systems of the apparatus. One set of junctions of each system is located on the guard ring, as shown at the upper left.

of the power supplied.

Method of attaching thermocouples. To attach the thermocouples to the hot plate, guard ring, and top shield, holes were drilled into these parts, and the junctions were cemented into them with "Technical G Copper Cement" obtained from the W. V. -B. Ames Company and used as directed. (See also Baker, Ryder, and Baker (5).) The thermocouples were of AWG 30 nylon-insulated wire and the junctions were fitted loosely in the holes before they were cemented. The cement is electrically insulating, but care is necessary to avoid contact between the junction and the walls of the hole. It was helpful to coat the junctions with a layer of copper cement, let it harden, and apply a coat of clear insulating varnish to the junctions before inserting them. The holes in the hot plate and guard ring were drilled from the top nearly to the bottom, so that the junctions would be as near as possible to the lower surface.

Method of installing heaters. Suitable circular grooves were cut in the upper surfaces of the hot plate, guard ring, and top shield to receive the various heater windings. The side of each groove nearest the axis is vertical and has a small lip at the top so that the windings will not slip out. As shown in Fig. 3, the hot plate contains two grooves, the guard ring one groove, and the top shield three.

The hot-plate heater is of AWG 32 glass-insulated Advance wire (4.76 ohms per ft). The radial distances of the grooves from the center of the plate, and the number of ohms wound into each groove were computed to give a high degree of hot-plate temperature uniformity. The windings in the two grooves are connected in series.

The guard-ring and top-shield heaters are of the same AWG 32 Advance wire as that used for the hot-plate heater. The windings in the three grooves of the top shield were series-connected. All windings were coated with lacquer to help hold them in place and to promote heat transfer. The resistances of the various heaters are shown in Figs. 4 and 5.

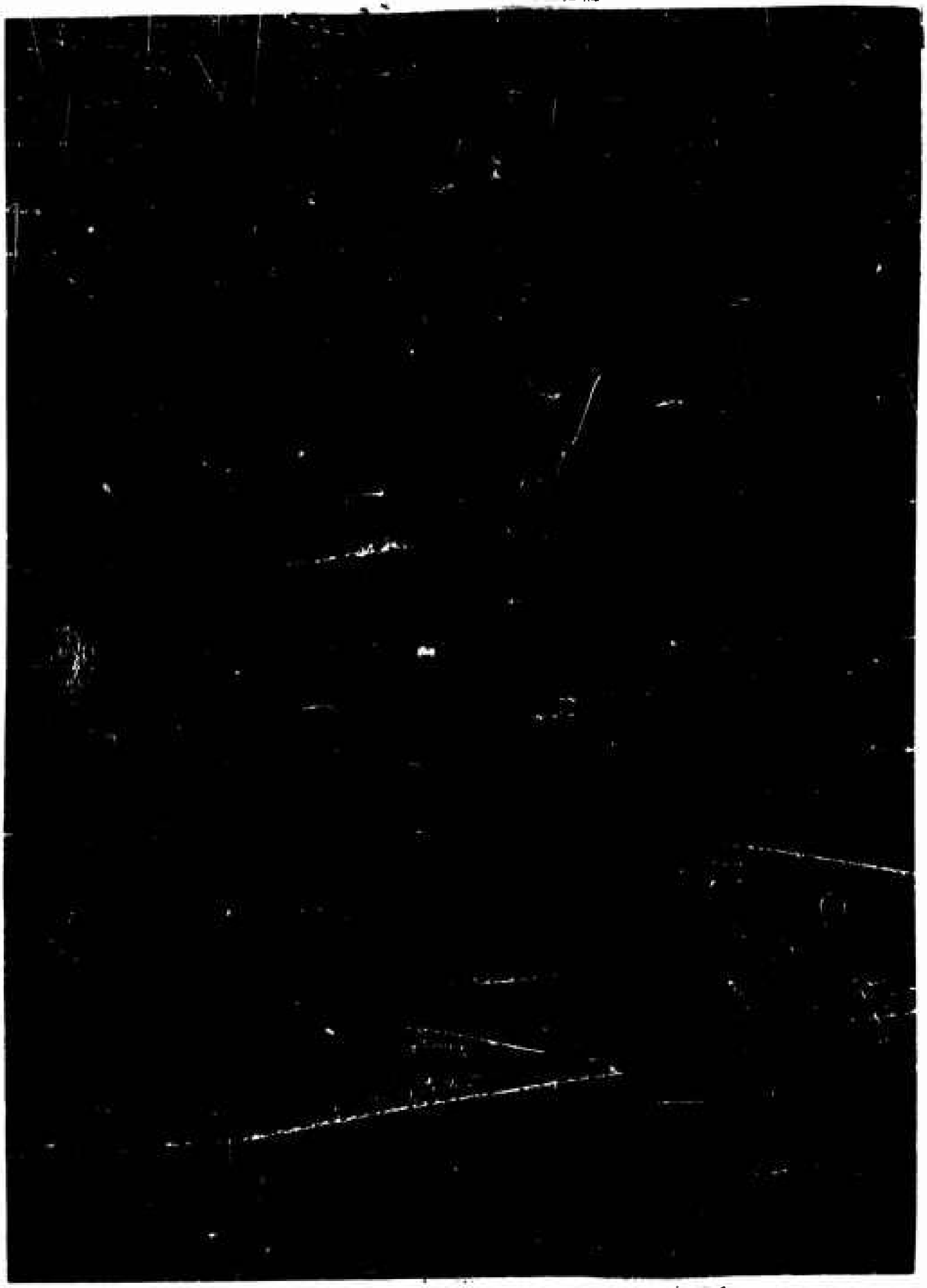
Electrical connecting wires. The wires with which the thermocouples and heaters are connected with the external equipment are brought approximately to the temperature of the hot plate before any of them make contact with the hot plate or the guard ring. The connecting wires enter the sample chamber through a metal tube with an Apiezon wax seal at the upper end.

Within the sample chamber the connecting wires are bound together in a cable. Some wires leave the cable to connect to the top-shield heater and (when needed) to connect to the strain gages. The other wires enter the space between the top shield and the guard ring through a slot cut in the downward-projecting lip of the top shield. They are then wound for one turn on a tempering groove cut for the purpose in the lower side of the top shield. Thermal contact with the top shield is promoted by coating the wires with Glyptal lacquer. From the tempering groove the wires run to the various heaters and thermocouple junctions where they are needed.

For testing the efficacy of the tempering there are two thermocouples on the cable of connecting wires: one before it passes around the tempering groove, the other after it passes around the tempering groove. There is also a heater wrapped on the cable, with which the first thermocouple can be brought to the desired temperature of the hot-plate assembly. A few runs were made using the heater and observing the two thermocouples, but unless unusual accuracy is required this is unnecessary. It is probable that the tempering groove is long enough but that the wires produce a slightly colder region on the top shield where the wires first come in contact with it.

As shown schematically in Fig. 6, the thermopile with which ΔT is measured requires 6 copper and 6 constantan wires, running from the guard ring to the water bath that establishes the cold-plate temperature. These wires run from the guard ring to the tempering ring on the top shield, pass through the slot in the top shield, and then are bound into a separate cable. This cable does not leave the sample chamber with the wires that run to the measuring equipment, but runs to a separate exit tube. This tube is bent into an inverted U so that after leaving the top of the sample chamber it turns downward to enter the water bath. The submerged end of the tube is closed, so that the tube is in effect a gas-tight extension of the sample chamber. The cold junctions are located near the closed end of the tube, imbedded in paraffin to improve thermal contact with the water bath. The tube is moderately flexible, and when measurements are being made it is bent so that the end containing the junctions is near and slightly below the cold plate. Some trouble was experienced in making the final soldered joint in the tube containing the thermopile. The thermocouple wires were nylon-insulated, and the soldering operation sometimes melted the insulation and caused the wires to short. Cotton insulation would probably be more practical in this particular application.

Water bath. The water bath in which the lower part of the sample chamber is immersed is a commercial thermostated, stirred bath. The cylindrical glass tank has an inside diameter of about 15 inches



and a depth of about 12 inches. Bath temperature oscillates by perhaps 0.1° F with the thermostating cycle, but the average temperature remains quite steady. When the highest accuracy is desired, the cycle of temperature control is analyzed, and ΔT is read just as the bath temperature passes through its average value. Note that the bath temperature need not be known with great accuracy provided it remains constant.

Refrigerating unit. The water bath in which the cold plate of the apparatus is immersed must be run at a temperature of about 95° F (35° C) in order to dissipate all heat to its surroundings without artificial cooling. In order to be able to work at cold-plate temperatures below 95° F, a laboratory refrigerating unit is used. This unit cools a built-in tank of stirred liquid (water and antifreeze). It has an auxiliary pump, which pumps cold liquid from the tank through a copper coil immersed in the water bath and then returns it to the tank of the refrigerating unit. A slight excess of cooling is supplied to the water bath, so that its thermostatic control can operate in the usual way.

The use of the laboratory refrigerating unit makes it possible to operate the apparatus with the cold-plate temperature as low as 41° F (5° C). The highest cold-plate temperature so far used is about 100° F (37.8° C), but considerably higher values should be attainable. A cold-plate temperature of 71.6° F (22° C) has been used for a large proportion of the measurements made thus far.

Control of gas in sample chamber. The sample chamber is connected to a vacuum system which can reduce the pressure below atmospheric as desired; the chamber can also be completely evacuated. Besides the line leading to the vacuum system there is a second line through which gases may be admitted to the sample chamber. The use of two lines permits the chamber to be continuously flushed when a new gas is introduced.

Two lines are also useful when gas has to be pumped off continuously, a situation that can occur when one is measuring a sample that gives off gas by desorption. By attaching the pressure gage to one line and the vacuum pump to the other, the true pressure in the chamber can be read without including any error resulting from the drop in the pumping line.

Pressures lower than 1 mm Hg are read with a Stokes (McLeod) gage, pressures between 1 and 50 mm Hg with an oil manometer, and pressures above 50 mm Hg with a mercury manometer.

c. Operating procedure

A run requires one working day. The sample is installed and the cold plate is immersed in the water bath before the day of the run. The approach to the steady state is begun early in the morning on the day of the run, by turning on the hot-plate, guard-ring, and top-shield heaters, the water-bath thermostatic control, and the laboratory refrigerating unit.

The hot and cold plates are brought as quickly as practicable to the desired temperatures, usually with a temperature difference of about 40° F (22.2° C) between the two plates. In several runs this temperature difference (ΔT) was initially brought to a value about 6 percent higher than the value at which measurements were to be made. It was held at this higher value for about 45 minutes; then it was dropped to the desired value by lowering the hot-plate temperature. This procedure hastens the approach to the steady state.

After the hot and cold plates and the top shield have reached the neighborhood of their desired operating temperature, a period of the run begins in which ΔT is held constant, or nearly so, by adjusting the hot-plate power. The guard ring is held automatically, and the top shield manually, at the same temperature as the hot plate. The changes required in hot-plate power gradually diminish as the steady state is approached.

At about 3 hours after the run is started, the adjustment of hot-plate power is discontinued, and the rating period begins. It is unwise to change the power input during this period, because the changes in power may make it difficult to recognize when the steady state has been reached. The rating period is continued to the end of the day, and measurements of the thermal conductivity are made at 30-minute intervals. At some convenient time during the run, sample thickness is measured. The pressure in the sample chamber is also read and recorded, unless the run is being made at atmospheric pressure.

The ASTM standard for guarded hot plates (1) requires that "tests shall be continued...until successive observations made at intervals not greater than 1 hour, over a period of 5 hours, give thermal conductivity values that are constant to within 1 percent." Inspection of the results of several runs convinced us that for our apparatus the 5-hour period was unnecessarily long. The criteria adopted for an acceptable set of data are given below:

(1) Retained readings (a "reading" is any one of the thermal-conductivity values observed during the rating period) must form a continuous group covering the time interval from the first acceptable reading to the last reading of the day. No reading made within this time interval may be excluded. The retained readings are averaged to give the thermal conductivity value.

(2) The accepted set of readings must contain no value that is more than 1 percent larger than the smallest.

(3) The accepted set of readings must cover a period of at least 3 hours. Exceptions to this rule may be made after due study, but must be noted when the data are reported.

(4) If the data cover a period of 5 hours or more, all readings in the last 5 hours will be used and no more.

2. Method of computing thermal conductivity

The thermal conductivity k is computed from the usual formula

$$k = \frac{\dot{Q}/A}{\Delta T/\Delta x}$$

wherein $\dot{Q}$ is the power supplied to the hot-plate heater, A is the effective area of the hot plate (9.52 in.^{2} = 61.43 cm^2), ΔT is the temperature difference between the two faces of the sample, and Δx is the sample thickness.

The value of $\dot{Q}$ obtained from the potentiometer readings is in absolute watts. For conversion to Btu per hour this value is multiplied by 3.4144. When the value of k in engineering units (Btu in/°F ft^{2}hr) has been found, it may be multiplied by the factor 3.4448 x 10^{-4} to convert it to scientific units (cal cm/°C cm^{2}sec).

3. Measurements on an NBS-calibrated material

A fibrous glass insulating board sold under the trade name of Aerocor has been adopted as a standard sample to be used for checking the apparatus. The National Bureau of Standards suggested the use of insulating board of this composition, and a specimen was sent to NBS for measurement of its thermal conductivity.

A number of runs were made in our laboratory on this material, and the results of a typical run are given in the following tabulation:

Run 25 (this laboratory)

Density, as tested, lb/ft^3	5.17
Thickness, as tested, inch	0.964
Thermal conductivity, Btu in./°F ft^2hr	0.233
Mean temperature of specimen, °F	115.0
Temperature gradient in specimen, °F/inch	41.5

For comparison, the test results obtained at the National Bureau of Standards on the sample that we submitted to them are given below. This sample was of the same material as that used in Run 25 but was not part of the same shipment from the manufacturer. The NBS report is dated 22 May 1959.

NBS Report

Test No.	HC51559	HC51859
Density, as tested, lb/ft^3	6.18	6.18
Thickness, as tested, inch	0.558	0.559
Thermal conductivity, Btu/hr ft^2 (°F/inch)	0.239	0.214
Mean temperature of specimens, °F	125.5	69.0
Temp. gradient in specimens, °F/inch	61.4	61.9

For a more direct comparison between our results and those of the NBS report, it is necessary to interpolate between the two mean temperatures of the NBS report, to obtain a value at 115.0 °F. Linear interpolation may be expected to be adequate and yields a thermal conductivity of 0.234 as compared with our value of 0.233. The difference is less than 1/2 percent, but such good agreement must be assumed to be partly fortuitous. The density of the sample measured at NBS was 20 percent greater than the density of our sample, which could in itself account for a difference in results of as much as 2 percent. It is planned to repeat the comparison using a sample from the same batt that the NBS-calibrated sample was taken from.

An accuracy of 1 percent in thermal-conductivity measurement is adequate for our present and anticipated experimental programs. Results obtained thus far indicate that this accuracy is obtainable.

4. Acknowledgments

We wish to thank Mr. Joseph P. Ciccolo and Mr. Malcolm N. Pilsworth, Jr. of this Center for assistance in this project. Mr. M. L. Clevett, Jr. formerly of this Center also contributed during the early stages of the work.

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